

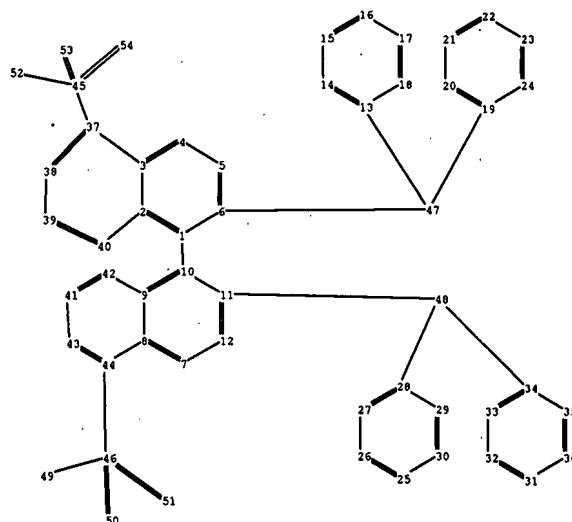
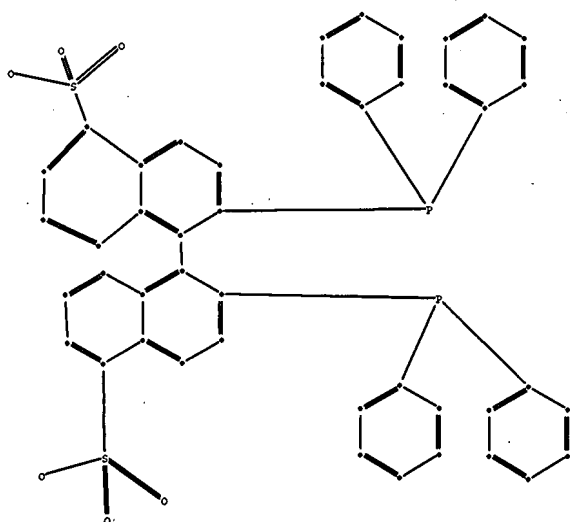
EAST Search History

Ref #	Hits	Search Query	DBs	Default Operator	Plurals	Time Stamp
S1	2	WO-9522405-\$.did.	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT	OR	ON	2007/08/03 18:52
S2	1603	(562/606,401).CCLS.	US-PGPUB; USPAT; USOCR	OR	OFF	2007/08/03 18:53
S3	2	EP-544455-\$.did.	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT	OR	ON	2007/08/06 10:07
S4	30	S2 and asymmetric adj hydrogenat\$	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT	OR	ON	2007/08/03 18:55
S5	6	("5563295").URPN.	USPAT	OR	ON	2007/08/03 19:01
S6	8	("5827794").URPN.	USPAT	OR	ON	2007/08/03 19:02
S7	2	("5563290").URPN.	USPAT	OR	ON	2007/08/03 19:06
S8	3117	binap	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT	OR	ON	2007/08/03 19:18
S9	182	binap.clm.	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT	OR	ON	2007/08/03 19:18
S10	2	sul??onated adj binap	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT	OR	ON	2007/08/03 19:33
S11	7	S9 and S2	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT	OR	ON	2007/08/03 19:33
S12	2	("5563290").URPN.	USPAT	OR	ON	2007/08/06 08:59
S13	1	("5274146").PN.	US-PGPUB; USPAT; USOCR	OR	OFF	2007/08/06 09:30

EAST Search History

S14	12	("5274146").URPN.	USPAT	OR	ON	2007/08/06 09:43
S15	1	("5274146").PN.	US-PGPUB; USPAT; USOCR	OR	OFF	2007/08/06 11:03
S16	2	JP-2005255544-\$.did.	US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT	OR	ON	2007/08/06 11:06
S17	2	((("5274146") or ("5324861"))).PN.	US-PGPUB; USPAT; USOCR	OR	OFF	2007/08/06 11:07
S18	7	("5324861").URPN.	USPAT	OR	ON	2007/08/06 11:08

We 8/6/07



chain nodes :

45 46 47 48 49 50 51 52 53 54

ring nodes :

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28
29 30 31 32 33 34 35 36 37 38 39 40 41 42 43 44

chain bonds :

1-10 6-47 11-48 13-47 19-47 28-48 34-48 37-45 44-46 45-52 45-53 45-54 46-49 46-50 46-51

ring bonds :

1-2 1-6 2-3 2-40 3-4 3-37 4-5 5-6 7-8 7-12 8-9 8-44 9-10 9-42 10-11 11-12 13-14 13-18
14-15 15-16 16-17 17-18 19-20 19-24 20-21 21-22 22-23 23-24 25-26 25-30 26-27 27-28
28-29 29-30 31-32 31-36 32-33 33-34 34-35 35-36 37-38 38-39 39-40 41-42 41-43 43-44

exact/norm bonds :

37-45 44-46 45-52 45-53 45-54 46-49 46-50 46-51

exact bonds :

1-10 6-47 11-48 13-47 19-47 28-48 34-48

normalized bonds :

1-2 1-6 2-3 2-40 3-4 3-37 4-5 5-6 7-8 7-12 8-9 8-44 9-10 9-42 10-11 11-12 13-14 13-18
14-15 15-16 16-17 17-18 19-20 19-24 20-21 21-22 22-23 23-24 25-26 25-30 26-27 27-28
28-29 29-30 31-32 31-36 32-33 33-34 34-35 35-36 37-38 38-39 39-40 41-42 41-43 43-44

Match level :

1:Atom 2:Atom 3:Atom 4:Atom 5:Atom 6:Atom 7:Atom 8:Atom 9:Atom 10:Atom 11:Atom 12:Atom
13:Atom

14:Atom 15:Atom 16:Atom 17:Atom 18:Atom 19:Atom 20:Atom 21:Atom 22:Atom 23:Atom
24:Atom 25:Atom 26:Atom 27:Atom 28:Atom 29:Atom 30:Atom 31:Atom 32:Atom 33:Atom 34:Atom
35:Atom 36:Atom 37:Atom 38:Atom 39:Atom 40:Atom 41:Atom 42:Atom 43:Atom 44:Atom 45:CLASS
46:CLAS\$47:Atom 48:Atom 49:CLAS\$50:CLAS\$51:CLAS\$52:CLAS\$53:CLAS\$54:CLASS

hve
8/6/2007

=> d his

(FILE 'HOME' ENTERED AT 11:00:05 ON 06 AUG 2007)

FILE 'REGISTRY' ENTERED AT 11:00:44 ON 06 AUG 2007

L1 STRUCTURE UPLOADED

L2 0 S L1

L3 4 S L1 SSS FULL

FILE 'HCAPLUS' ENTERED AT 11:02:15 ON 06 AUG 2007

L4 2 S L3

FILE 'STNGUIDE' ENTERED AT 11:03:26 ON 06 AUG 2007

FILE 'HCAPLUS' ENTERED AT 11:10:42 ON 06 AUG 2007

E US2006-211882/APPS

L5 0 S US2006-211882/APPS

L6 1 S US2006-211882/AP, PRN, PN

E L6 RN

FILE 'REGISTRY' ENTERED AT 11:12:14 ON 06 AUG 2007

L7 10 S 150273-68-0/RN OR 775352-14-2/RN OR 7732-18-5/RN OR 80-59-1/R

FILE 'HCAPLUS' ENTERED AT 11:15:00 ON 06 AUG 2007

L8 2 S 150273-68-0/RN OR 775352-14-2/RN

L9 3 S AMANO,A?/AU AND IGARASHI,D?/AU AND SAYO,N?/AU

Mr. Smith
8/6/07

=>

Uploading C:\Program Files\Stnexp\Queries\2007 cases\10550564\core str.str

L1 STRUCTURE UPLOADED

=> d l1

L1 HAS NO ANSWERS

L1 STR

* STRUCTURE DIAGRAM TOO LARGE FOR DISPLAY - AVAILABLE VIA OFFLINE PRINT *

Structure attributes must be viewed using STN Express query preparation.

=> s l1

SAMPLE SEARCH INITIATED 11:01:21 FILE 'REGISTRY'

SAMPLE SCREEN SEARCH COMPLETED - 2 TO ITERATE

100.0% PROCESSED 2 ITERATIONS

0 ANSWERS

SEARCH TIME: 00.00.01

FULL FILE PROJECTIONS: ONLINE **COMPLETE**

BATCH **COMPLETE**

PROJECTED ITERATIONS: 2 TO 124

PROJECTED ANSWERS: 0 TO 0

L2 0 SEA SSS SAM L1 *←*

=> s l1 sss full

FULL SEARCH INITIATED 11:01:36 FILE 'REGISTRY'

FULL SCREEN SEARCH COMPLETED - 71 TO ITERATE

100.0% PROCESSED 71 ITERATIONS

4 ANSWERS

SEARCH TIME: 00.00.01

L3 4 SEA SSS FUL L1 *←*

=> d l3 1-4 ide

L3 ANSWER 1 OF 4 REGISTRY COPYRIGHT 2007 ACS on STN

RN 865062-26-6 REGISTRY

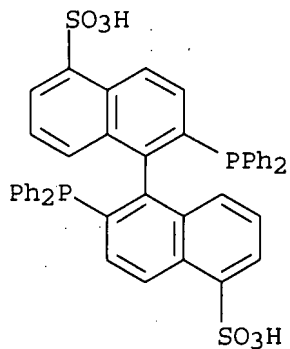
ED Entered STN: 11 Oct 2005

CN [1,1'-Binaphthalene]-5,5'-disulfonic acid, 2,2'-bis(diphenylphosphino)-
(9CI) (CA INDEX NAME)

MF C44 H32 O6 P2 S2

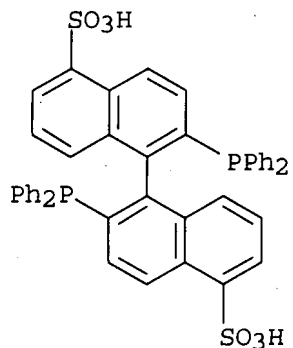
CI COM

SR CA



PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

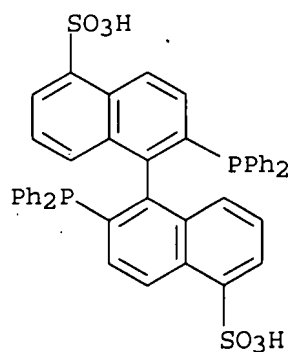
L3 ANSWER 2 OF 4 REGISTRY COPYRIGHT 2007 ACS on STN
 RN 864956-92-3 REGISTRY
 ED Entered STN: 11 Oct 2005
 CN [1,1'-Binaphthalene]-5,5'-disulfonic acid, 2,2'-bis(diphenylphosphino)-,
 disodium salt (9CI) (CA INDEX NAME)
 MF C44 H32 O6 P2 S2 . 2 Na
 SR CA
 LC STN Files: CA, CAPLUS
 CRN (865062-26-6)



● 2 Na

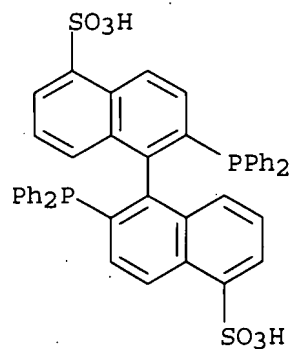
1 REFERENCES IN FILE CA (1907 TO DATE)
 1 REFERENCES IN FILE CAPLUS (1907 TO DATE)

L3 ANSWER 3 OF 4 REGISTRY COPYRIGHT 2007 ACS on STN
 RN 39341-59-4 REGISTRY
 ED Entered STN: 05 Sep 2004
 CN [1,1'-Binaphthalene]-5,5'-disulfonic acid, 2,2'-bis(diphenylphosphino)-,
 (R)- (9CI) (CA INDEX NAME)
 MF C44 H32 O6 P2 S2
 CI COM
 SR CA



PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

L3 ANSWER 4 OF 4 REGISTRY COPYRIGHT 2007 ACS on STN
 RN 150271-78-6 REGISTRY
 ED Entered STN: 23 Sep 1993
 CN [1,1'-Binaphthalene]-5,5'-disulfonic acid, 2,2'-bis(diphenylphosphino)-,
 disodium salt, (R)- (9CI) (CA INDEX NAME)
 MF C44 H32 O6 P2 S2 . 2 Na
 SR CA
 LC STN Files: CA, CAPLUS, USPATFULL
 CRN (739341-59-4)



● 2 Na

1 REFERENCES IN FILE CA (1907 TO DATE)
 1 REFERENCES IN FILE CAPLUS (1907 TO DATE)

=> fil hcaplus
 COST IN U.S. DOLLARS
 FULL ESTIMATED COST

SINCE FILE	TOTAL
ENTRY	SESSION
180.80	181.01

10/550564 METHOD FOR PRODUCING OPTICALLY ACTIVE CARBOXYLIC

FILE 'HCAPLUS' ENTERED AT 11:02:15 ON 06 AUG 2007
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*Reg# Search
 huc 8/6/07*

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FILE COVERS 1907 - 6 Aug 2007 VOL 147 ISS 7
 FILE LAST UPDATED: 5 Aug 2007 (20070805/ED)

New CAS Information Use Policies, enter HELP USAGETERMS for details.

This file contains CAS Registry Numbers for easy and accurate substance identification.

=> s 13

L4 2 L3 *X*

=> d 14 1-2 ibib abs

L4 ANSWER 1 OF 2 HCAPLUS COPYRIGHT 2007 ACS on STN
 ACCESSION NUMBER: 2005:1020733 HCAPLUS
 DOCUMENT NUMBER: 143:306189
 TITLE: Preparation of pyridinecarboxamides with recyclable catalysts and without the use of halogenation agents
 INVENTOR(S): Shimazu, Hidetaka; Tamashima, Tomoyuki
 PATENT ASSIGNEE(S): Koei Chemical Co., Ltd., Japan
 SOURCE: Jpn. Kokai Tokkyo Koho, 18 pp.
 CODEN: JKXXAF
 DOCUMENT TYPE: Patent
 LANGUAGE: Japanese
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2005255544	A	20050922	JP 2004-65682	20040309
PRIORITY APPLN. INFO.:			JP 2004-65682	20040309

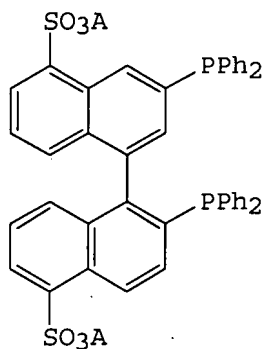
AB Pyridinecarboxamides are prepared by isomerization of pyridinealoximes in multiphase solvent mixts. in the presence of (A) mixts. of hydrophilic phosphines and transition metals, or (B) water-soluble complexes comprising the phosphines and metals. Thus, 4-pyridinealoxime was refluxed with sulfonated BINAP and RuCl₂(cod) in 1-butyl-4-methylimidazolium PF₆ salt and C₆H₆ for 24 h, then the ionic liquid was recovered, which was used in the same reaction 4 more times. Total yield of 4-pyridinecarboxamide was 94.5%.

L4 ANSWER 2 OF 2 HCAPLUS COPYRIGHT 2007 ACS on STN
 ACCESSION NUMBER: 1993:581016 HCAPLUS

10/550564 METHOD FOR PRODUCING OPTICALLY ACTIVE CARBOXYLIC

DOCUMENT NUMBER: 119:181016
 TITLE: Preparation of water-soluble alkali metal sulfonate-substituted binaphthylphosphine transition metal complexes and enantioselective hydrogenation method using them
 INVENTOR(S): Ishizaki, Takerou; Kumobayashi, Hidenori
 PATENT ASSIGNEE(S): Takasago International Corp., Japan
 SOURCE: Eur. Pat. Appl., 9 pp.
 CODEN: EPXXDW
 DOCUMENT TYPE: Patent
 LANGUAGE: English
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 544455	A1	19930602	EP 1992-310561	19921119
EP 544455	B1	19970212		
R: CH, DE, FR, GB, IT, LI				
JP 05170780	A	19930709	JP 1991-331535	19911121
JP 2736947	B2	19980408		
US 5274146	A	19931228	US 1992-977638	19921117
US 5324861	A	19940628	US 1993-116583	19930907
PRIORITY APPLN. INFO.:			JP 1991-331535	A 19911121
			US 1992-977638	A3 19921117
OTHER SOURCE(S):		CASREACT 119:181016; MARPAT 119:181016		
GI				



AB [M(X)_n(Q)(SO₃A-BINAP)]Y (M = Ru, Ir, Rh, Pd, etc.; SO₃A-BINAP = tertiary phosphine represented by formula I (A = alkali metal atom), X = Cl, Br, iodo; n = 0, 1; Q = benzene or p-cymene, Y = Cl, Br, iodo, ClO₄, PF₆, BF₄) were prepared and shown to be catalysts for the enantioselective hydrogenation of olefins, ketones, and imines.

=> fil stng

COST IN U.S. DOLLARS

FULL ESTIMATED COST

SINCE FILE

ENTRY

10.86

TOTAL

SESSION

191.87

10/550564 METHOD FOR PRODUCING OPTICALLY ACTIVE CARBOXYLIC

DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS)

SINCE FILE
ENTRY

TOTAL
SESSION

CA SUBSCRIBER PRICE

-1.56

-1.56

FILE 'STNGUIDE' ENTERED AT 11:03:26 ON 06 AUG 2007

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FILE CONTAINS CURRENT INFORMATION.

LAST RELOADED: Aug 3, 2007 (20070803/UP).

<http://www.cas.org/support/stngen/stndoc/properties.html>

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1 150273-68-0/RN
1 775352-14-2/RN
1 7732-18-5/RN
1 80-59-1/RN
1 1333-74-0/RN
1 5309-52-4/RN
1 16957-70-3/RN
1 32231-50-8/RN
1 49642-47-9/RN
1 56006-48-5/RN

L7 10 150273-68-0/RN OR 775352-14-2/RN OR 7732-18-5/RN OR 80-59-1/RN
OR 1333-74-0/RN OR 5309-52-4/RN OR 16957-70-3/RN OR 32231-50-8/R
N OR 49642-47-9/RN OR 56006-48-5/RN

=> d 17 ide 1-10

L7 ANSWER 1 OF 10 REGISTRY COPYRIGHT 2007 ACS on STN

RN 775352-14-2 REGISTRY

ED Entered STN: 05 Nov 2004

CN Ruthenate(1-), [(1R)-2,2'-bis(diphenylphosphino-κP)[1,1'-binaphthalene]-5,5'-disulfonato(2-)]chloro[(1,2,3,4,5,6-η)-1-methyl-4-(1-methylethyl)benzene]-, sodium chloride (1:2:1) (9CI) (CA INDEX NAME)

MF C54 H44 Cl O6 P2 Ru S2 . Cl . 2 Na

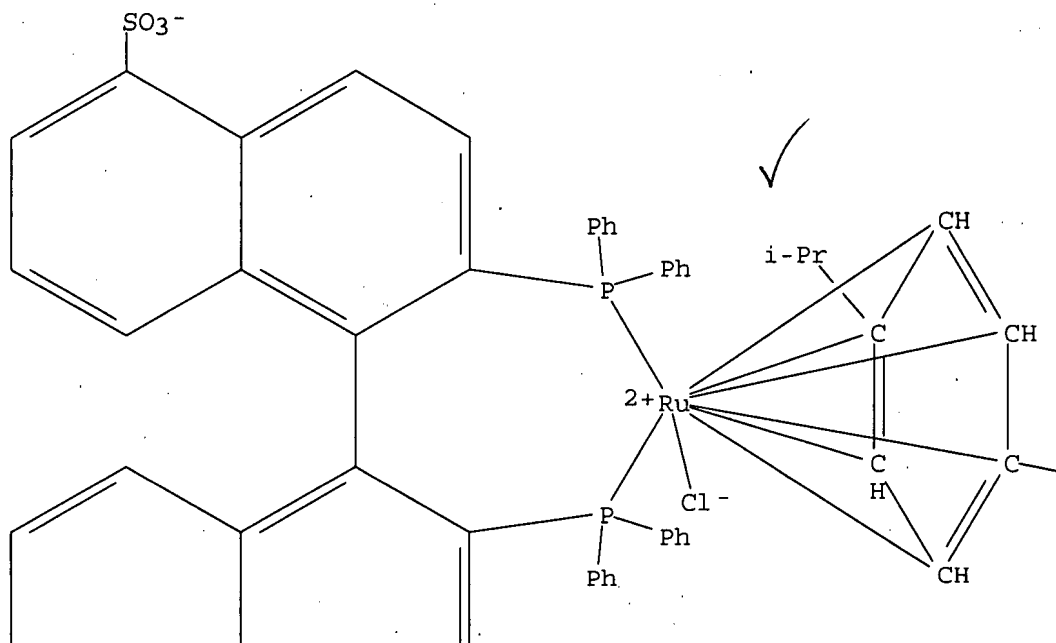
CI CCS

SR CA

LC STN Files: CA, CAPLUS, CASREACT, USPATFULL

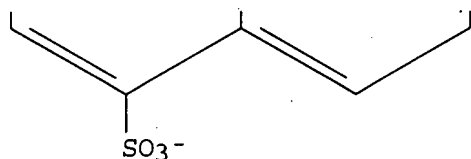
CRN (795269-49-7)

PAGE 1-A



PAGE 1-B

Me



PAGE 2-A

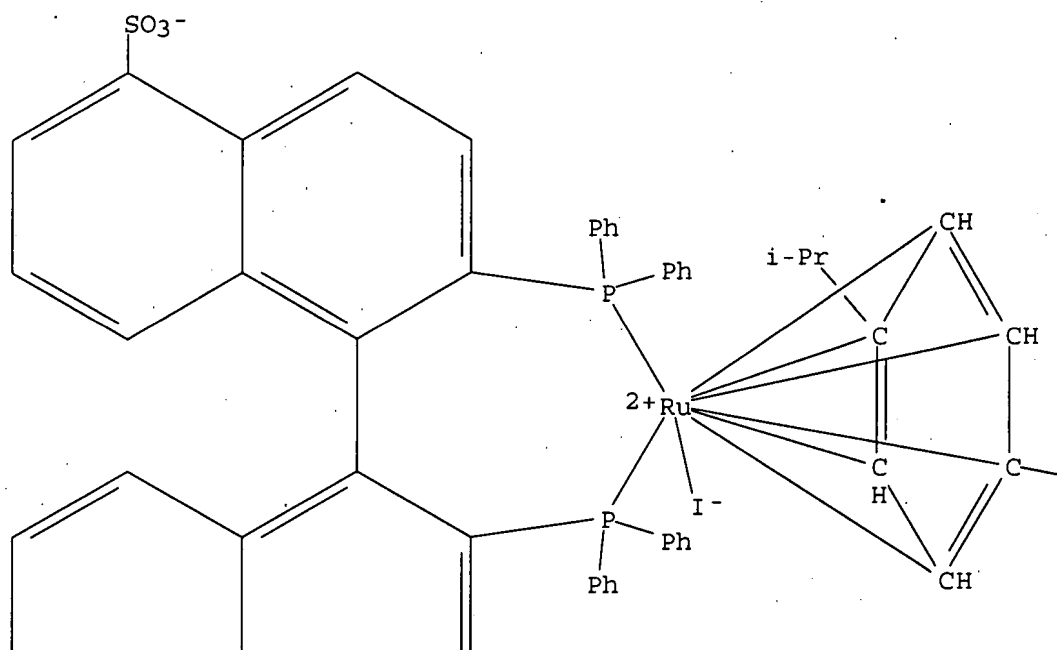
● Cl⁻

● 2 Na⁺

1 REFERENCES IN FILE CA (1907 TO DATE)
1 REFERENCES IN FILE CAPLUS (1907 TO DATE)

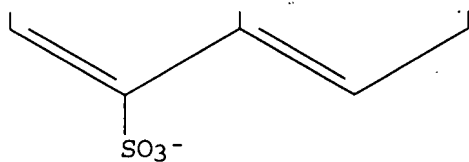
L7 ANSWER 2 OF 10 REGISTRY COPYRIGHT 2007 ACS on STN
RN 150273-68-0 REGISTRY
ED Entered STN: 23 Sep 1993
CN Ruthenate(1-), [2,2'-bis(diphenylphosphino)[1,1'-binaphthalene]-5,5'-
disulfonato(2-)-P,P']iodo[(1,2,3,4,5,6-η)-1-methyl-4-(1-
methylethyl)benzene]-, sodium iodide (1:2:1), (R)- (9CI) (CA INDEX NAME)
MF C54 H44 I O6 P2 Ru S2 . I . 2 Na
CI CCS
SR CA
LC STN Files: CA, CAPLUS, CASREACT, USPATFULL
CRN (762212-66-8)

PAGE 1-A



PAGE 1-B

—Me



● I^-

● 2 Na^+

2 REFERENCES IN FILE CA (1907 TO DATE)
2 REFERENCES IN FILE CAPLUS (1907 TO DATE)

10/550564 METHOD FOR PRODUCING OPTICALLY ACTIVE CARBOXYLIC

=> s 150273-68-0/RN or 775352-14-2/RN

2 150273-68-0

0 150273-68-0D

2 150273-68-0/RN

(150273-68-0 (NOTL) 150273-68-0D)

1 775352-14-2

0 775352-14-2D

1 775352-14-2/RN

(775352-14-2 (NOTL) 775352-14-2D)

L8

2 150273-68-0/RN OR 775352-14-2/RN

=> d 18 ibib abs

L8 ANSWER 1 OF 2 HCAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 2004:857540 HCAPLUS

DOCUMENT NUMBER: 141:349812

TITLE: Stereoselective catalytic hydrogenation process for producing optically active carboxylic acids from α,β -unsaturated carboxylic acids

INVENTOR(S): Amano, Akira; Igarashi, Daisuke; Sayo, Noboru

PATENT ASSIGNEE(S): Takasago International Corporation, Japan

SOURCE: PCT Int. Appl., 29 pp.

CODEN: PIXXD2

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2004087632	A1	20041014	WO 2004-JP4373	20040326
W:	AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW			
RW:	BW, GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG			
GB 2414987	A	20051214	GB 2005-19756	20040326
GB 2414987	B	20061025		
CN 1753857	A	20060329	CN 2004-80004883	20040326
JP 2006521371	T	20060921	JP 2006-507695	20040326
US 2006211882	A1	20060921	US 2005-550564	20050926
IN 2005CN02777	A	20070608	IN 2005-CN2777	20051027
PRIORITY APPLN. INFO.:			JP 2003-89605	A 20030328
			WO 2004-JP4373	W 20040326

OTHER SOURCE(S): CASREACT 141:349812; MARPAT 141:349812

AB A hydrogenation method is described for producing an optically active carboxylic acid [e.g., (2R)-methylbutanoic acid] with a high optical purity is obtained by the hydrogenation of the corresponding α,β -unsatd. carboxylic acid (e.g., tiglic acid) using a chiral ruthenium sulfonated BINAP hydrogenation catalyst [e.g., [RuI[p-cymene][(R)-(SO₃Na)₂BINAP]]I] complex; the complex catalyst used

10/550564 METHOD FOR PRODUCING OPTICALLY ACTIVE CARBOXYLIC

can be recovered and reused as an aqueous solution

REFERENCE COUNT: 2 THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS
RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

10/550564 METHOD FOR PRODUCING OPTICALLY ACTIVE CARBOXYLIC

Inventor Search
Aug 8/07

FILE 'REGISTRY' ENTERED AT 11:12:14 ON 06 AUG 2007

L7 10 S 150273-68-0/RN OR 775352-14-2/RN OR 7732-18-5/RN OR 80-59-1/R

FILE 'HCAPLUS' ENTERED AT 11:15:00 ON 06 AUG 2007

L8 2 S 150273-68-0/RN OR 775352-14-2/RN

=> s amano,a?/au and igarashi,d?/au and sayo,n?/au

416 AMANO,A?/AU

30 IGARASHI,D?/AU

107 SAYO,N?/AU

L9 3 AMANO,A?/AU AND IGARASHI,D?/AU AND SAYO,N?/AU

=> d 19 1-3 ibib abs

L9 ANSWER 1 OF 3 HCAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 2005:1130650 HCAPLUS

DOCUMENT NUMBER: 143:405612

TITLE: Preparation of chiral phosphines, transition metal complexes containing the same as the ligand, and process for production of optically active carboxylic acids

INVENTOR(S): Amano, Akira; Igarashi, Daisuke; Sayo, Noboru

PATENT ASSIGNEE(S): Takasago International Corporation, Japan

SOURCE: PCT Int. Appl., 27 pp.

CODEN: PIXXD2

DOCUMENT TYPE: Patent

LANGUAGE: Japanese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2005097811	A1	20051020	WO 2005-JP3117	20050225
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RW:	BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG			
US 2007060772	A1	20070315	US 2006-540166	20060929
PRIORITY APPLN. INFO.:			JP 2004-97508	A 20040330
			WO 2005-JP3117	A1 20050225

OTHER SOURCE(S): MARPAT 143:405612

GI

* STRUCTURE DIAGRAM TOO LARGE FOR DISPLAY - AVAILABLE VIA OFFLINE PRINT *

AB Sulfonated phosphines represented by the general formula (I) (wherein X1 =

10/550564 METHOD FOR PRODUCING OPTICALLY ACTIVE CARBOXYLIC

O, CH₂; X₂ = CH₂, CH₂CH₂, CH₂CH₂CH₂, 1,2-dimethylethylene, isopropylidene, difluoromethylene; A = a Group IA alkali metal of the periodic table, hydrogen, an ammonium ion; a, b, c, d = 0 or 1, with the proviso that the cases wherein the sum of a, b, c, and d is 0 are excepted) are prepared. Desired optically active carboxylic acids of formula R₁R₂C*HC*HR₃CO₂H [* denotes an asym. carbon atom; R₁-R₃ = H, optionally branched cycloalkyl, each (un)substituted aromatic hydrocarbyl or heterocyclyl, acyloxy, acylamino, alkoxy, aryloxy, alkoxy-carbonyl, CO₂H, furyloxy, thienyloxy; or R₁ and R₂ or R₁ and R₃ = (CH₂)_m-X₃-(CH₂)_n (X₃ = CH₂, N, O, S); m = 1,2; n = an integer of 0-3] are prepared from a carboxylic acid having a carbon-carbon double bond of formula R₁R₂C:CR₃CO₂H (R₁-R₃ = same as above) through asym. hydrogenation with a catalyst consisting of a transition metal complex containing a water-soluble ligand I. The water soluble ligand I permits easy separation of the used catalyst from the product by liquid-liquid separation alone and enables the recovery of an expensive transition metal and the reuse of the catalyst. Thus, 2.52 g (R)-5,6,7,8,5',6',7',8'-octahydro-2,2'-bis(diphenylphosphino)-1,1'-binaphthyl [(R)-H₈-BINAP] was added to 3.68 g concentrated H₂SO₄ at 0-5°, stirred, followed by adding 35.8 g 30% SO₃-H₂SO₄ via a syringe, and the mixture was warmed to room temperature, stirred for 1 h, stored at 5° for 2 wk, added slowly to crushed ice (200 g), neutralized by adding dropwise 50% NaOH at ≤15°, distilled to completely remove water under reduced pressure to give a solid (62 g). The solid was stirred with 140 mL 10% aqueous MeOH solution for 1 h, filtered to remove sodium sulfate, and concentrated to give 3.2 g (R)-SO₃Na-H₈-BINAP (II) [a+b+c+d = 2 (32%), a+b+c+d = 3 (53%)]. II (0.13 g), 38.3 mg [RuCl₂(p-cymene)]₂, and 3 mL MeOH were mixed and stirred at room temperature for 24 h, followed evaporation of the solvent to give 0.19 g 0.17 mg [RuCl((R)-SO₃Na-H₈-BINAP)(p-cymene)]Cl (III). Tiglinic acid (3.3 g), 2.42 mg III, 4 mL diisopropyl ether, and 3 mL distilled water were heated at 80° and H pressure of 2.5 MPa for 2 h in an autoclave to give 2.9 g (R)-2-methylbutanoic acid (96.4% ee).

REFERENCE COUNT: 6 THERE ARE 6 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L9 ANSWER 2 OF 3 HCAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 2005:1075754 HCAPLUS

DOCUMENT NUMBER: 143:378576

TITLE: Preparation of optically active transition metal/diamine complex and process for producing optically active alcohol with the complex

INVENTOR(S): Amano, Akira; Igarashi, Daisuke; Sayo, Noboru

PATENT ASSIGNEE(S): Takasago International Corporation, Japan

SOURCE: PCT Int. Appl., 64 pp.

CODEN: PIXXD2

DOCUMENT TYPE: Patent

LANGUAGE: Japanese

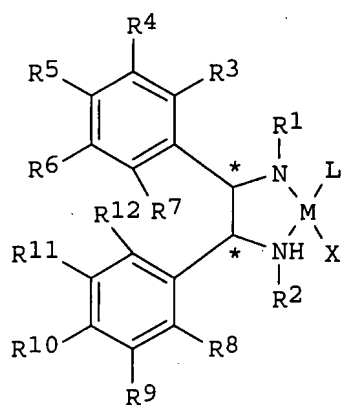
FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

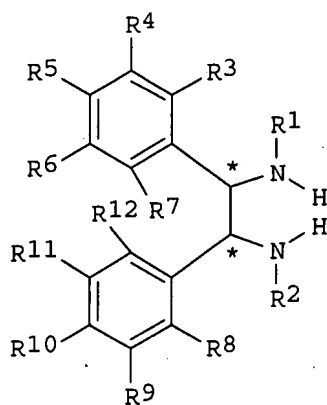
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2005092830	A1	20051006	WO 2005-JP5728	20050328
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD,				

GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SM, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW
 RW: BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

US 2007149831 A1 20070628 US 2006-594744 20060929
 PRIORITY APPLN. INFO.: JP 2004-96472 A 20040329
 WO 2005-JP5728 W 20050328
 OTHER SOURCE(S): MARPAT 143:378576
 GI



I



II

AB A water-soluble, optically active transition metal/diamine complex represented by the general formula (I) [wherein R1 and R2 each represents hydrogen, a hydrocarbon group, -SO2R13 (wherein R13 = optionally substituted hydrocarbon group, substituted amino, camphoryl); R3 to R12 each represents hydrogen, an each optionally substituted hydrocarbon group, alkoxy, aryloxy, or aralkyloxy or substituted amino; M represents a transition metal; X represents halogeno; L represents a ligand; and * indicates asym. carbon; provided that at least one of R3 to R7 and R8 to R12 is substituted amino] and an optically active diamine compound (II) (R2-R13) constituting the ligand of the complex are prepared. The optically active transition metal/diamine complex is useful as a catalyst for asym. synthesis and can be easily separated from reaction products through liquid separation, etc. and is recyclable. A process for producing an optically active alc. comprises using the catalyst I for asym. reduction of a ketone. Thus, a solution of 26.76 g 4-dimethylaminobenzaldehyde in 80 mL THF was added to a mixture of 10.4 g hydrazine sulfate, 78 mL H2O, and 28% aqueous NH3 at ≤40° over 2 h, stirred at ≤40° for 2-3 h, treated with 28% aqueous NH3 to make the aqueous layer alkali, treated with 100 mL toluene, cooled to 10°, and filtered to give 19.75 g 4,4'-bis(dimethylamino)benzazine (III) (83.9%). A mixture of Zn powder (19.6 g) and 300 mL THF was treated dropwise with 28.45 g TiCl4 III at -40° over 40 min at ≤40°, stirred at the same temperature for 30 min and then at -30 to -25° for 1 h, treated with 8.82 g III at -25°, stirred for 3 h while the temperature was raised to room temperature,

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and left to stand overnight to give, after workup, crude 1,2-bis[4-(dimethylamino)phenyl]ethane-1,2-diamine (IV). IV (3.48 g) was converted into the HCl salt and then back into racemic free amine (1.04 g) which was separated twice by HPLC using a Chiralcel OD-H column to give 63.1 mg (1R,2R)-1,2-bis[4-(dimethylamino)phenyl]ethane-1,2-diamine which (50 mg) was mixed with 17.7 mg Et₃N in 1 mL CH₂Cl₂, treated with portionwise with a solution of 28.1 mg benzenesulfonyl chloride in 1 mL CH₂Cl₂ under ice-cooling, and stirred at the same temperature for 1 h to give, after purification

by TLC, 57.4 mg (1R,2R)-N-(benzenesulfonyl)-1,2-bis[4-(dimethylamino)phenyl]ethane-1,2-diamine (V). A mixture of V (4 mg), 1.86 mg [RuCl₂(mesitylene)]₂, 0.45 g sodium formate, and 4 mL H₂O was treated with 0.2 g acetophenone, stirred at 50° for 2.5 h to give 0.19 g (R)-1-phenethyl alc. (optical purity 90.78% ee).

REFERENCE COUNT: 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L9 ANSWER 3 OF 3 HCAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 2004:857540 HCAPLUS

DOCUMENT NUMBER: 141:349812

TITLE: Stereoselective catalytic hydrogenation process for producing optically active carboxylic acids from α,β -unsaturated carboxylic acids

INVENTOR(S): Amano, Akira; Igarashi, Daisuke; Sayo, Noboru

PATENT ASSIGNEE(S): Takasago International Corporation, Japan

SOURCE: PCT Int. Appl., 29 pp.

CODEN: PIXXD2

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2004087632	A1	20041014	WO 2004-JP4373	20040326
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW				
RW: BW, GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
GB 2414987	A	20051214	GB 2005-19756	20040326
GB 2414987	B	20061025		
CN 1753857	A	20060329	CN 2004-80004883	20040326
JP 2006521371	T	20060921	JP 2006-507695	20040326
US 2006211882	A1	20060921	US 2005-550564	20050926
IN 2005CN02777	A	20070608	IN 2005-CN2777	20051027
PRIORITY APPLN. INFO.:			JP 2003-89605	A 20030328
			WO 2004-JP4373	W 20040326

OTHER SOURCE(S): CASREACT 141:349812; MARPAT 141:349812

AB A hydrogenation method is described for producing an optically active carboxylic acid [e.g., (2R)-methylbutanoic acid] with a high optical

purity is obtained by the hydrogenation of the corresponding α,β -unsatd. carboxylic acid (e.g., tiglic acid) using a chiral ruthenium sulfonated BINAP hydrogenation catalyst [e.g., $[\text{RuI}[\text{p-cymene}][(\text{R})-(\text{SO}_3\text{Na})_2\text{BINAP}]]\text{I}$] complex; the complex catalyst used can be recovered and reused as an aqueous solution

REFERENCE COUNT: 2 THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT